California Environmental Protection Agency

Air Resources Board

Engineering and Laboratory Branch
Monitoring and Laboratory Division

MLD SOP ES01

STANDARD OPERATING PROCEDURE FOR THE TOTAL VOLATILE MEASUREMENT OF CONSUMER PRODUCTS

March 10, 1998, Revision 2

DISCLAIMER: Mention of any trade name or commercial product in Method 310 and associated Standard Operating Procedures does not constitute endorsement or recommendation of this product by the Air Resources Board. Specific brand names and instrument descriptions listed in the Standard Operating Procedures are equipment used by the ARB laboratory. Any functionally equivalent instrumentation can be used.

1 INTRODUCTION

This document describes a procedure for the measurement of the weight percent total volatile content in the non-propellant portion of a consumer product as determined by U.S. EPA Method 24/24A, and ASTM D2369-87. The mention of trade names or commercial products in this Standard Operating Procedure does not constitute endorsement or recommendation by the Air Resources Board (ARB) and are included as examples only.

2 SUMMARY OF METHOD

This procedure measures the total volatile content of a given consumer product. A known amount of sample is weighed into a dish, which is placed in a forced draft oven for 60 minutes at 110°C. The difference in sample weight, before and after heating, is considered the total volatile material for the sample tested.

3 INTERFERENCE/LIMITATION

3.1 Certain consumer products may react with the aluminum dishes. In these cases, substitute with Teflon dishes.

3.2 Products containing polymeric materials may exhibit weight loss not considered "volatile."

3.3 Substances considered "low vapor pressure" as defined in the consumer products regulation may volatilize under the conditions of this analysis. The weight percent total volatile content must be adjusted for this loss.

4 APPARATUS

4.1 Oven, forced draft, able to maintain a temperature of 110 ± 5°C (ASTM Type II A or Type II B recommended).

4.2 Laboratory Fume Hood - explosion-proof recommended.

4.3 Analytical Balance, capacity of 100 g ± 0.0001 g.

4.4 Weighing Dishes

4.4.1 Aluminum Weighing Dishes, 58 mm x 18 mm w/ a smooth (planar) bottom surface (per ASTM 2369)

4.4.2 Teflon PFA Petri Dishes, 50 mm x 15 mm w/ a smooth (planar) bottom surface (per ASTM 2369)

4.5 Disposable Syringe, 3 - 5 mL, w/ caps

4.6 Desiccator

4.7 Wrist Action Shaker

4.8 Gloves, sharps resistant

4.9 Forceps

4.10 Scribe

5 REAGENTS

5.1 Water, distilled, ASTM Type I

5.2 Acetone, reagent grade

6 PROCEDURE

6.1 Scribe aluminum dish with identification number - two dishes are required per sample. Place aluminum dishes in oven for 60 minutes, at 110°C (these can be prepared up to 24 hours ahead of time). Allow to cool to ambient temperature in desiccator.

NOTE 1 - Gloves shall be worn during all steps of this procedure to prevent weighing errors due to handling.

6.2 Obtain an aluminum dish from desiccator with forceps. Weigh and record the empty aluminum dish weight, to the nearest 0.1 mg.

6.3 Vortex sample the homogeneity, using a syringe, withdraw approx. 3 mL. Cap and weigh syringe with liquid sample. Dispense approximately 1 gm of liquid sample into the aluminum dish. Cap and reweigh the syringe after sampling. Prepare replicate.

NOTE 2 - If the sample in the dish does not form a thin film, add 2 mL of acetone, and evenly disperse.

6.4 Put the dish with sample in the oven at 110°C for one hour. Cool in desiccator to room temperature. Record the weight of the aluminum dish with residue.

NOTE 3 - If the sample appears to be reacting with the aluminum dish, a Teflon dish can be substituted.

6.5 Calculate weight percent total volatile matter of the sample in accordance with Section 8. If results of the duplicate weight percents differ by more than 0.5 % volatile material, repeat Section 6.

7 QUALITY CONTROL

7.1 The balance is calibrated daily, using the internal calibration program on the balance.

7.2 After the balance is calibrated it is checked using a 1.0 g ASTM Class 1 mass. The value is recorded on the control charts. The 1.0 g weight should be within $\pm$ 3s of the expected value.

7.3 A control check for the gravimetric analysis is made by preparing a sample of ASTM Type 1 water and carrying it through the procedure. If the oven is operating properly, the water sample will be calculated as 100% VOC. Record the value on the control chart. The value should be $\pm$ 3s of the calculated value.

7.4 In addition a sample of known concentration designated as the Trip sample is carried through the analysis.

7.5 If the balance weight or the water gravimetric analysis are not within the control values, check the conditions and reanalyze. The balance should be calibrated semi - annually by a certified technician.

8 CALCULATIONS

8.1 Data Summary

8.1.1 Weight of empty aluminum dish to the nearest 0.1 mg, as determined by Section 6.2 - [A]

8.1.2 Weight of syringe w/ liquid sample to the nearest 0.1 mg, as determined by Section 6.3 - [B]

8.1.3 Weight of syringe after dispensing sample to the nearest 0.1 mg, as determined by Section 6.3 - [C]

8.1.4 Weight of cooled aluminum dish w/ sample residue to the nearest 0.1 mg, as determined by Section 6.4 - [D]

8.2 Equations

8.2.1 Weight (gm) of Liquid Sample = [B] - [C]

8.2.2 Weight (gm) of Residue = [D] - [A]

8.2.3 Total Volatile Sample, weight fraction = $\frac{([B] - [C]) - ([D] - [A])}{([B] - [C])}$

8.2.4 Rel % Diff. = $\frac{(Dup\ 1) - (Dup\ 2)}{[(Dup\ 1) + (Dup2)]/2} \times 100\%$

Appendix A

Gravimetric Determination of Consumer Product Samples

1. The Sartorius MC1 analytical balance is used for the gravimetric analysis. The instrument is to be calibrated on a daily basis. Press F1 on the balance and the instrument will do an internal calibration.

2. A check of the accuracy of the balance is made using an ASTM Class 1 mass. Zero the balance. Using the forceps, remove the 1.0g ASTM Class 1 Calibration Mass and place on the balance, wait for the reading to give a stable reading for 15 seconds and record the weight on the daily balance check log to 5 places and initial (Figure 1). Record the value on the balance control chart, date, and initial as shown in Figure 2. If the balance is not in the control limits, then the balance should be recalibrated as described in #1 above. After the calibration, re-weigh the ASTM mass. The balance is calibrated by an outside contractor on an annual basis.

3. Prior to doing the gravimetric analysis, place some of the aluminum weigh dishes in the oven for 60 minutes at 110°, remove the dishes from the oven and allow them to cool to ambient temperature in the desiccator. This will "dry" the dishes.

4. Scribe a weighing dish with an identification number/mark- two dishes are required per sample. The dishes are either aluminum foil (57 mm diameter x 10 mm high) with a flat bottom or teflon PFA petri dish (58 mm diameter x 15 mm high) with a flat bottom. The teflon will be used in cases where the product may react with the aluminum dish.

5. Prepare the gravimetric analysis data sheet (Figure 3). The lab id # is in the far left column, adjacent is the replicate dish identification (either a letter or a number). Each sample has 2 dishes assigned to it. These are the sample replicates. In addition there is the water control (confirms the operation of the oven) and the Trip sample, these are also done in replicate and treated exactly like samples.

6. With forceps, remove from the desiccator one of the weighing dishes. Place on the analytical balance, weigh and record the weight of the empty dish to 0.1 mg. See the gravimetric data sheet for properly filling out the information (Figure 3a).

7. Pre-weigh all the empty weigh dishes and record on the data sheet. Be certain you have indicated the ten samples and the designated duplicate, as well as the Trip sample and a water control.

8. Mix the sample aliquot thoroughly either by shaking, vortexing or placing the sample on a platform shaker.

9. Using a 3 mL syringe, withdraw approximately 3 mL of the product, wipe excess with Kimwipe. Cap and weigh the syringe with the liquid sample, placing the syringe plunger down in a tared 150 mL beaker. Record the weight in the column marked syringe initial weight. Dispense approximately 1.0 mL of the sample into the weigh dish. Cap and reweigh the syringe. Record the weight in the column syringe final weight. The difference is the actual amount of the product placed in the weigh dish.

10. Reweigh the amount remaining in the syringe, this will be the syringe initial weight for the sample replicate. Record that value for the next weigh dish.

11. Dispense approximately 1.0 mL of the sample into the weigh dish as described in #9 above. Record the weight as the syringe final weight. The difference is the actual amount of the product in the dish. Discard the syringe.

12. If the sample is too viscous (ex. solid or gel) use a transfer tube. Draw up about 1.0 g product, wipe outsides with a Kimwipe, and dispense directly into the tared dish. Record the weight in the column marked weight sample. Mark diagonally through the syringe weight columns and indicate that transfer tube was used.

13. All the weigh dishes with the sample in them are placed in the oven at 110°C for 60 minutes. It is important that this time does not exceed $\pm$ 5 minutes. The weigh dish and sample are removed and placed in the desiccator to cool to room temperature. Each weigh dish is then reweighed, with the sample residue and noted in the column weight pan + residue.

14. Any observations regarding the sample residue should be noted in the analyst's notebook.

15. When all the information is recorded, including the water control and the Trip sample, draw a line diagonally through any unused portions of the data sheet as shown in Figure 3b.

16. Calculations:
    a. Weight of sample = (syringe initial weight) - (syringe final weight)
    b. Weight of residue = (weight of dish w/ sample residue) - (weight of empty dish)
    c. % volatile (w/w) = 100 x $\frac{\text{[(weight sample) - (weight residue)]}}{\text{weight sample}}$

17. If the results between the two analysis differ by more than 1%, repeat the analysis.

****NOTES****

**Gloves must be worn during all steps of this procedure where weights or weighed objects are dealt with.

**If the sample in the dish does not form a thin film, add 2 mL of distilled water, toluene, or methanol and evenly disperse.

**Aluminum dishes can be used for most products. However where there may be a reaction with the aluminum, teflon dishes can be substituted.

**DO NOT EXCEED THE TIME FOR THE SAMPLES TO STAY IN THE OVEN!!

Gravimetric Analysis

The gravimetric analysis is for the determination of <u>total</u> volatile material in a sample. The procedure is based on ASTM D2369-87. An aliquot of product is weighed into an aluminum foil dish and heated in a forced-air oven at 110° for 60 mins. The total volatile material is the difference in weight of the sample before and after heating. The total voc is subsequently corrected for non-voc, low vapor pressure and exempt compounds in the final weight % calculation.

Calculations:

Weight fraction, Total VOC = (weight sample - weight residue) / weight sample

SOP REVISION HISTORY

1. October 10, 1996: Additions to the QC section, the addition of the trip sample and clarify the calibration of the balance.

2. March 10, 1998. Adjusted document font to Times New Roman 12. Inserted appendix A formerly a stand-alone document.